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Research Article

Chitosan Concentration in Aqueous Media via Electrical Conductivity

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Abstract

Chitosan is the deacetylated polymer of chitin. Chitosan has many advantageous properties such as biodegradability, biocompatibility, nontoxicity, and hydrophilicity. It is also antioxidant, antimicrobial, renewable, and low-cost. The properties and potential uses of chitosan have been of great interest to researchers for many years. Chitosan has limited solubility in neutral and alkali solutions. Many methods to quantify the chitosan concentration have been proposed i.e. UV–vis spectrophotometry, fluorspectrophotometry, cathodic stripping voltammetry, High-performance Liquid Chromatography (HPLC), and resonance Rayleigh scattering method. This paper proposed a practical non-invasive method for determining chitosan content in low-pH aqueous media.

Introduction

Chitosan is a linear biopolymer mostly synthesized by deacetylation processes from chitin. It contains units of D-glycosamide and D-glucosamine joined by ß-(1,4) linkages (Figure 1). The exoskeleton of squid and crab shells is the most well-known source of chitin. In recent times, it has also been extracted from insects and fungi. Chitosan's diverse applications and unique properties—such as low immunogenicity, biocompatibility, biodegradability, and non-toxicity, make it one of the most common natural polymers in the world [1].

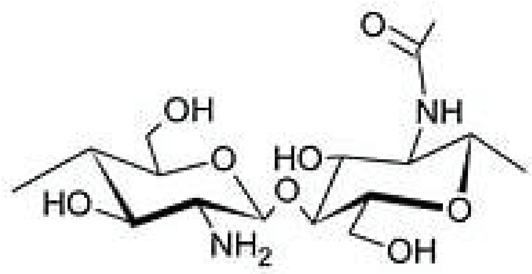

Figure 1: The molecular structure of chitosan.

Numerous industries utilize chitosan, including biomedicine, cosmetics, wastewater treatment, agriculture, and the food sector [2]. Chitosan has been a promising biomaterial for the development of controlled drug delivery systems [3], wound healing management [4], effective antibacterial and antiviral applications [5], and scaffolds for bone and tissue regeneration [6] because of its biocompatibility, antimicrobial activity, and bioactivity [7]. Chitosan is also of interest as a gene therapy vector [8].

Because of its reactive hydroxyl and amino groups, which provide excessive hydrogen bonding and a positive charge, chitosan is a weak base soluble in aqueous acidic media. This creates highly viscous solutions ideal for forming free films and coatings and a unique linear polycation with a high charge density. These films can be filled with nanoparticles [9].

The charged chitosan, the only polycation found in nature, is influenced by the media's pH and level of acetylation. The degree of acetylation and molecular weight determine its solubility. Chitosan, having a higher molecular weight, is only soluble in acidic aqueous solutions at higher levels of deacetylation. The chitosan backbone contains plenty of protonated NH_2 units, which makes it soluble in acidic aqueous

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solutions. It has a pKa value of about 6.5. About 50% of all amino groups of chitosan must be protonated for chitosan to become soluble [10]. Temperature, degree of acetylation, pH, and thermophilicity are other variables that impact chitosan solubility.

Chitosan exhibits poor solubility at neutral and basic pH levels. Therefore, several chitosan derivatives have been produced with enhanced solubility [11].

There are many options for analyzing chitosan including UV–vis spectrophotometry [12,13], fluorspectrophotometry [2], cathodic stripping voltammetry [14], high-performance liquid chromatography [15], and resonance Rayleigh scattering method [16] but the advantage of measuring electrical conductivity is that it is non-invasive and allows us to monitor the kinetics of the process, such as adsorption. A fast, non-invasive detection method to quantify chitosan concentration at low pH aqueous solutions was proposed in this study.

Materials

Chitosan derived from squid (Glentham Life Sciences, UK) MW=580000 Da, degree of deacetylation > 90%, acetic acid (99%, Penta, Czech Republic), sodium hydroxide (98%, Penta, Czech Republic), and deionized water were applied for the samples.

Methods

Chitosan samples were completely dissolved in 1% (v/v) acetic acid solution. Chitosan concentrations ranged from 10 to 150 ppm. The final pH of the solution was measured as 2.65 using a pH meter (Inolab pH Level 2, Germany). The samples were analyzed for their electrical conductivity by a conductivity meter (Inolab Cond 7110, Germany) over the temperature range from 25 °C to 35 °C (Figure 2). The conductivity measurements were achieved by simply immersing the conductivity meter`s probe into the tubes containing different amounts of chitosan and waiting a couple of seconds until the conductivity reading stabilized. During the experiments, the samples were held in a water bath to ensure all the measurements were taken at a constant temperature (GFL 1083, Germany).

A higher pH, ranging from pH 3 to 5, caused some chitosan to be partially or completely insoluble. The experiments were also carried out using 1% (v/v) acetic acid solution, and the pH of the acetic acid solution was adjusted using 0.1M NaOH solution.

All measurements were carried out at least in two duplicates. The linear regression and statistical analysis were performed in Microsoft Excel 2016.

Result and discussion

The measurement is based on the fact that the resistance force of the environment acting on a given ion is proportional to the viscosity as stated in Stokes` Law:

$$F_d = -6\pi\eta r v \tag{1}$$

where; F_d, is the frictional force also known as Stokes' drag – acting on the interface between the fluid and the particle (N); η, is the dynamic viscosity (Pa.s); r, is the radius of the spherical object (m); v, is the flow velocity relative to the object (m/s).

Our measurements show a linear dependency of chitosan concentration on electrical conductivity (Figures 2,3) at pH 2.65 and 3. Chitosan was not fully soluble in acetic acid solutions adjusted to pH 3, 4, and 5 (for pH ≥ 3). Although the conductivity method is limited, an acidic environment is the easiest way to work with chitosan in a dissolved state. To have it in a dissolved state for pH higher or equal to 3, chitosan was first dissolved in acetic acid, then brought to the final pH, and conductivity trials were conducted. However, no linear relationship between chitosan concentration and conductivity was obtained at pH >3 because the electric forces of dissolved ions were much stronger than the viscosity effects. Too high pH causes the driving force of dissolved ions to be significantly higher than the resistance force, so the method is not usable for pH >3.

A high correlation coefficient (R^2) suggests that a large proportion of the variance in the dependent variable is predictable from the independent variables. This generally means the model fits the data well. It is observed that R^2 values decreased with rising temperature of the chitosan solution.

The positively charged chitosan molecules interact with negatively charged ions in the medium, probably forming

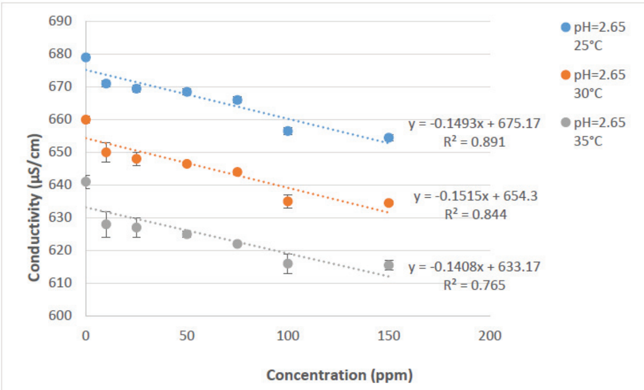

Figure 2: Electrical conductivity of chitosan in 1% acetic acid solution (pH 2.65).

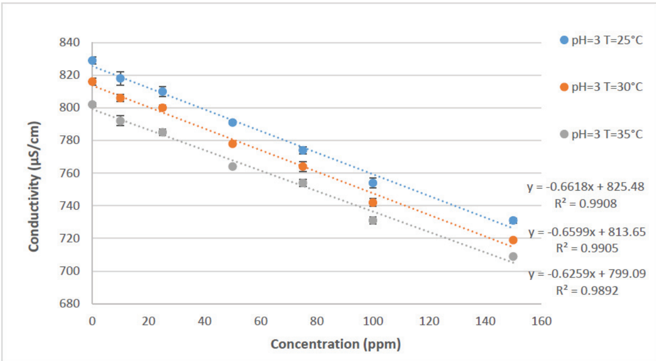

Figure 3: The electrical conductivity of chitosan samples dissolved in 1% (v/v) acetic acid solution (pH=3).

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a double layer. The influence of chitosan concentration on electrical conductivity is primarily due to changes in viscosity of the solution, thus decreasing the viscosity. Higher temperatures increase proton mobility but may lead to structural deterioration if excessive [17]. The degree of deacetylation and the presence of crosslinking agents or additives further affect chitosan's conductivity, with higher deacetylation typically resulting in greater positive charge density. Despite these influences, the exact mechanisms and extent of chitosan's effect on conductivity remain complex and require further research for definitive confirmation [18].

Conclusion

In conclusion, it would be appropriate to state that this analytical method to determine chitosan concentration based on viscosity measurement can be suitable for low pH (≤ 3) to ensure the solubility of chitosan. This simple, non-invasive method can be applied, for example, in the monitoring of adsorption processes. The influence of chitosan’s positive charge on conductivity was not confirmed. Further research is needed to fully understand and confirm these effects under different conditions and applications.

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